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APPLICATION OF RAPIDLY SOLIDIFIED SUPERALLOYS.(U)
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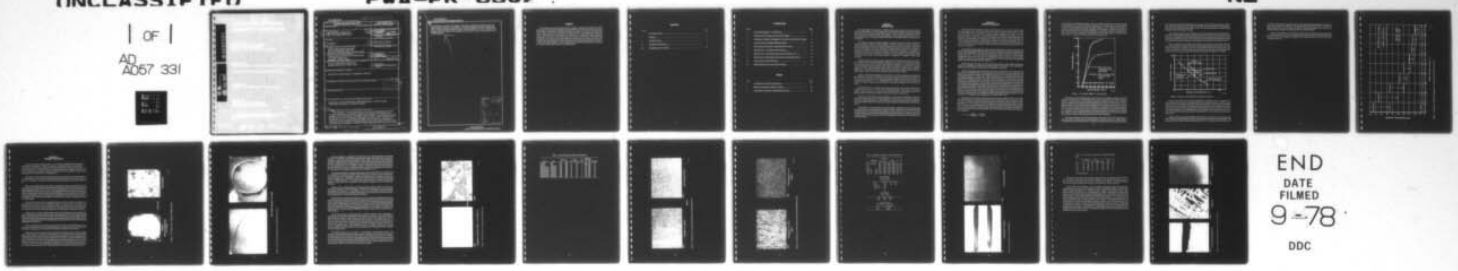

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This program is being conducted for the purpose of applying the principle of rapid solidification to superalloy powders and subsequent development of a stronger alloy composition for jet engine turbine airfoils. Centrifugal atomization and forced convective cooling are being used to produce the fast cooled material. During this period, a second generation high-speed turbine drive assembly for atomization was completed, and requirements for gas quenching through the use of swirl flow nozzles identified. Powders of IN100 and MAR M-200 were successfully

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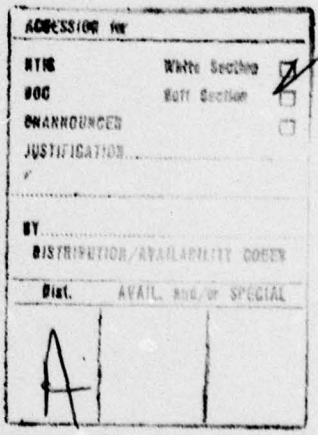
consolidated, extruded, and forged, and initial mechanical property tests show high-strength levels can be attained. New alloy studies have been started and laser surface melting to achieve locally high solidification rates on experimental samples has been added to the program as a means to screen many alloys on an expedited basis.



SUMMARY

This program is being conducted for the purpose of applying the principle of rapid solidification to superalloy powders and subsequent development of a stronger alloy composition for jet engine turbine airfoils. Centrifugal atomization and forced convective cooling are being used to produce the fast cooled material. During this period, a second generation high-speed turbine drive assembly for atomization was completed, and requirements for gas quenching through the use of swirl flow nozzles identified. Powders of IN100 and Mar M-200 were successfully consolidated, extruded, and forged, and initial mechanical property tests show high-strength levels can be attained. New alloy studies have been started and laser surface melting to achieve locally high solidification rates on experimental samples has been added to the program as a means to screen many alloys on an expedited basis.

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SECTION I
INTRODUCTION

The performance improvements of today's military gas turbine, such as the Pratt & Whitney Aircraft F100, over earlier engines were made possible through advancements in design technology and materials processing. Better alloys, by virtue of chemical composition, played only a minor role in achieving present day capability. Future engine projections, however, are demanding that better materials be developed in order that still higher levels of performance can be achieved.

The turbine module is especially dependent on improvements in such alloy properties as higher temperature capability, better stability, and better corrosion resistance. The alloys presently being used in this section were developed more than 15 years ago. It has not been that a lack of development interest has existed since then that these alloys are still in use. Rather, it has been the inability to improve the nature of alloying under conditions now imposed for subsequent processing and component fabrication. Precision casting alloy compositions are limited because of such constraints as crucible and mold interactions and massive phase occurrence. Forging alloys are limited because of constraints of segregation during ingot processing.

Superalloy powder metallurgy studies conducted at the P&WA/Florida facility have shown that the use of powder, particularly powder solidified at very high rates of cooling, can eliminate the constraints noted and can enable more effective alloying for the improvement of basic material properties. Several examples which support this statement are as follows. Chemical segregation in fast cooled superalloy powders can be controlled to a submicron level. Massive phases can be eliminated. Solubility of alloying elements can be extended without deleterious phase reaction. None of these can be achieved in ingot or precision casting.

Further, the inherent homogeneity of the powder is such that subsequent processing and heat treatment can be used very effectively to promote maximum material utilization. Abnormal grain growth, for example, can be achieved in superalloy powder materials for optimization of mechanical properties above $\frac{1}{2}$ T_M. MAR M-200 alloy powder, processed and reacted in this manner, is, in fact, stronger than, and as ductile as, the same composition cast in a directional mode.

P&WA/Florida has constructed a device that can produce metal powders solidified and cooled at rates in excess of 10^5 °C/sec. The underlying principle is forced convective cooling, whereby powder particles of controlled size are accelerated into a high thermal conductivity gaseous medium maintained at high ΔT between itself and the metal particle.

The purpose of this ARPA sponsored program is to refine the process mechanics used with the powder producing device for fast quenching bulk lots of powder and, subsequently, apply the technology of rapid solidification to the development of an alloy composition that is stronger than the existing MAR M-200 alloy and that can be implemented for the production of better turbine airfoils.

The program is a 40-month effort and is organized as a progression of events starting with a parametric study of the requirements necessary to achieve high yields of fast quenched powder and terminating in the fabrication and testing of turbine airfoils. This report is the fourth technical report and covers the 10th through 12th months of the program. It deals with the study of producing fast cooled powder and with the metallurgical and mechanical characteristics of resulting material.

SECTION II
PROCESS MECHANICS

The device used for production of fast cooled powder is one in which a central rotary atomizer disintegrates a molten metal stream into fine particles and accelerates them into a high mass-flow helium quench environment. Principal design parameters are based on a metal atomization rate of 0.15 kg/sec. It is presently capable of handling metal charges up to 23 kilograms (based on nickel). The unit has 3 annular gas nozzles which were sized to gas mass-flow and velocity commensurate with maintenance of a high ΔT on the basis of calculated heat flux profiles and particle trajectories. A radial impulse turbine is used for the atomizer drive. The unit is provided with conventional vacuum induction melting and tundish metering. The sequence of operations includes vacuum melting, Helium pressurization, pouring and atomization, and finally, gas quenching.

In the previous report period, the major activities with respect to process mechanics were directed toward analyzing repeatability of the combined operations, statistical characteristics of powder yield, and efficiency of the cross-flow helium gas quench for observed heat release profiles. During that time, it was found that the atomization could be carried out with excellent reproducibility from run-to-run, that the resulting powder yield was of a statistically Normal distribution, and that the cross-flow quench efficiency could be improved by incorporating additional nozzles and modifying total mass-flow.

During this period, the bulk of activity was directed primarily toward producing superalloy powder for consolidation, extrusion, and forging studies. Consequently, effort expended on additional process mechanics and the nature of operation was minimal.

Twenty runs with the device were made in the past 3 months. Most were made with the alloys IN-100 and MAR M-200, as reported previously. Five of the twenty runs were made with experimental compositions which are described in Section III. No operating difficulties were encountered during any of these trials. Likewise during these runs, no deviation from the now standardized operating procedures were made. With the exception of one of the last runs to be made with an experimental alloy, the powder yield stayed within the statistical boundaries described in the previous report. This particular one run is interesting, however, in that the yield of powder deviated significantly from all others. The alloy was a modified IN-100 composition to which tantalum was added at the expense of titanium and in which the carbon concentration was increased about 80% above normal. The results from this run with respect to yield are shown in figure 1 and are compared to the mean yield observed in all other runs to date made at these conditions. As can be seen for this singular instance, the mean particle size changed from 88 microns to 76 microns, and the total amount of material less than 105 microns (the maximum size of interest to this program) was increased from an average of 68% to 89%.

Specifically why this highly desirable result was obtained is unknown at this time. Other experimental alloys run in this period, as well as those run previously, showed no deviation of this type. Also, the empirical relationships dealing with rotary atomization, which we believe to hold reasonably true for our operation, would not suggest such a change unless some major variation in fluid viscosity occurred. Although this has not been checked, it seems highly doubtful in light of the results with other alloys. What appears most logical is that the coupling characteristics to the atomizer surface were altered appreciably, with the end effect being a change in constant in the relationship.

$$d = K \left(\frac{M^{0.2}}{r^{0.3}\,\omega^{0.6}}\right) \left(\frac{\sigma^{0.1}\mu^{0.2}}{\rho^{0.8}}\right)$$

in which the first paranthetical expression relates to operating conditions, the second to liquid metal properties. K is a constant related to coupling and, for wetted surfaces, is about equal to 1/3 on the basis of published reports for other rotary atomizers . A 14% variation in this value (88 microns to 76 microns) does not appear unrealistic for the change in conditions we are running, and it could be the result of some minor modification to the atomizer surface that heretofore has gone unnoticed. Where warranted in the subsequent months of this program, additional study will be given to the surface wetting characteristics during operation.

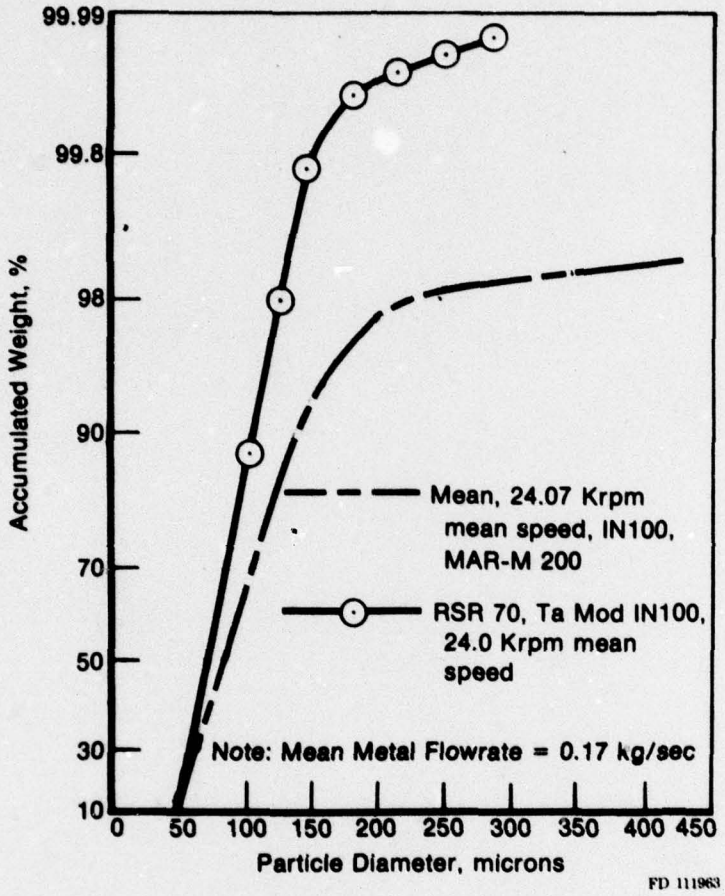

Figure 1. Accumulated Weight % vs Particle Size

Previously, it had been reported that a new atomizer drive assembly was being constructed which would produce twice the tangential velocity for atomization than now possible. This new assembly is a radial inflow turbine, capable of operating to 35,000 rpm with a 10.2 cm diameter surface, compared to the existing radial impulse turbine with a limit of 25,000 rpm for a 7.62 cm diameter surface. This assembly has been completed. The installation and checkout will require about a month of equipment down-time, and because it is felt that the more important purpose now is to produce material for evaluation, it has not been installed. It is expected that within 1 to 2 months from now an adequate amount of experimental material and time will be available for installation without interferring with the rest of the program work. Until that time, further direct studies of powder yield and distribution will be held in abeyance.

The original analyses of He quench requirements were based on an exclusive generation of 50μm particles with subsequent heat release, relative to location, velocity, etc., based solely on criteria associated with this particle size. In previously reported work, effort was directed toward

better definition of actual gas requirements on the basis of both cross-flow quenching, which is now used in the device, and on swirl-flow, for which earlier experimentation indicated higher efficiencies than obtainable on the present system. The cross-flow analysis was completed and reported in the previous quarterly report.

The swirl-flow analysis is also complete now. The results indicate that, for the given particle distribution and heat release profiles, cooling rates equal to those of the cross-flow mode can be achieved more efficiently (less total gas-flow) with the swirl configuration. Of equal, practical importance, preliminary layouts of the required swirl manifold-nozzle assembly indicate that maintainability and ease of cleaning are much better for swirl than for the cross-flow system.

Figure 2 illustrates the principle of gas mass-flow reduction for the swirl mode. The cross-flow system provides a constant axial velocity through each separately acting quench zone. The design circumstance, by necessity, must be set to the idealized axial velocity at the zone ID, that closest to the atomizer itself. Since the true requirement is one of continuously decreasing need, the OD velocity is excessively high, and results in unnecessary throughput in this region.

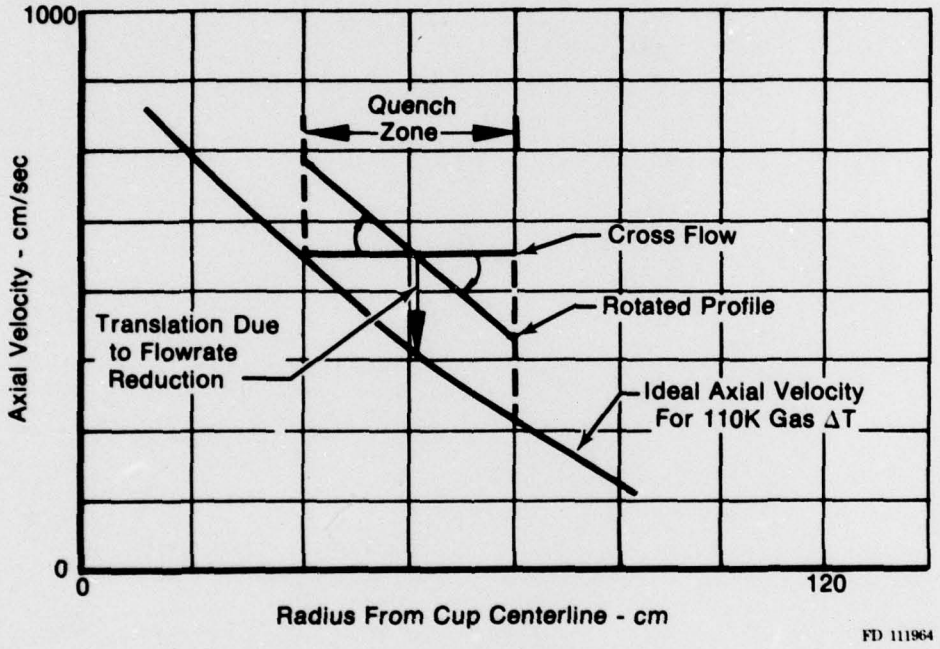

Figure 2. Schematic of Flow Reduction Using a Swirl System

On the other hand, the swirl-flow configuration incorporates vanes to produce a 3-dimensional gas flow field that is characterized by both tangential and axial velocities, both of which decrease with increasing radius. The rotated profile in figure 2 shows the result of changing from cross-flow to swirl without changing the total mass flowrate. The resulting axial velocity profile is everywhere parallel to and greater than the idealized profile. A translation of the swirl-flow profile downward to the ideal takes place by decreasing the overall zone flowrate. Thus, the swirl-flow configuration can provide particle cooling equivalent to the cross-flow system at a lower cooling flow.

Figure 3 shows the mass-flow difference between the two schemes on an equivalent zone basis. In the previously reported work on the cross-flow system, nine zones were determined to be about ideal for the present particle distribution, both from a standpoint of gas delivery and

practicality. The cross-flow requirement was 2.15 kg of gas per kg of metal atomized. For the swirl mode, this requirement is reduced to 1.47 kg of gas per kg of atomized metal, a reduction of approximately 32%. As can be seen from figure 3, the greatest savings occur closest to the atomizer, where the shape of the ideal axial velocity profile is steepest.

Because of better efficiency the swirl-flow concept is being used to size the gas recirculation system for the powder device. In turn, these data will be used to estimate the cost of operation for volume production. This analysis is scheduled to be completed in May of this year, and will be reported at that time.

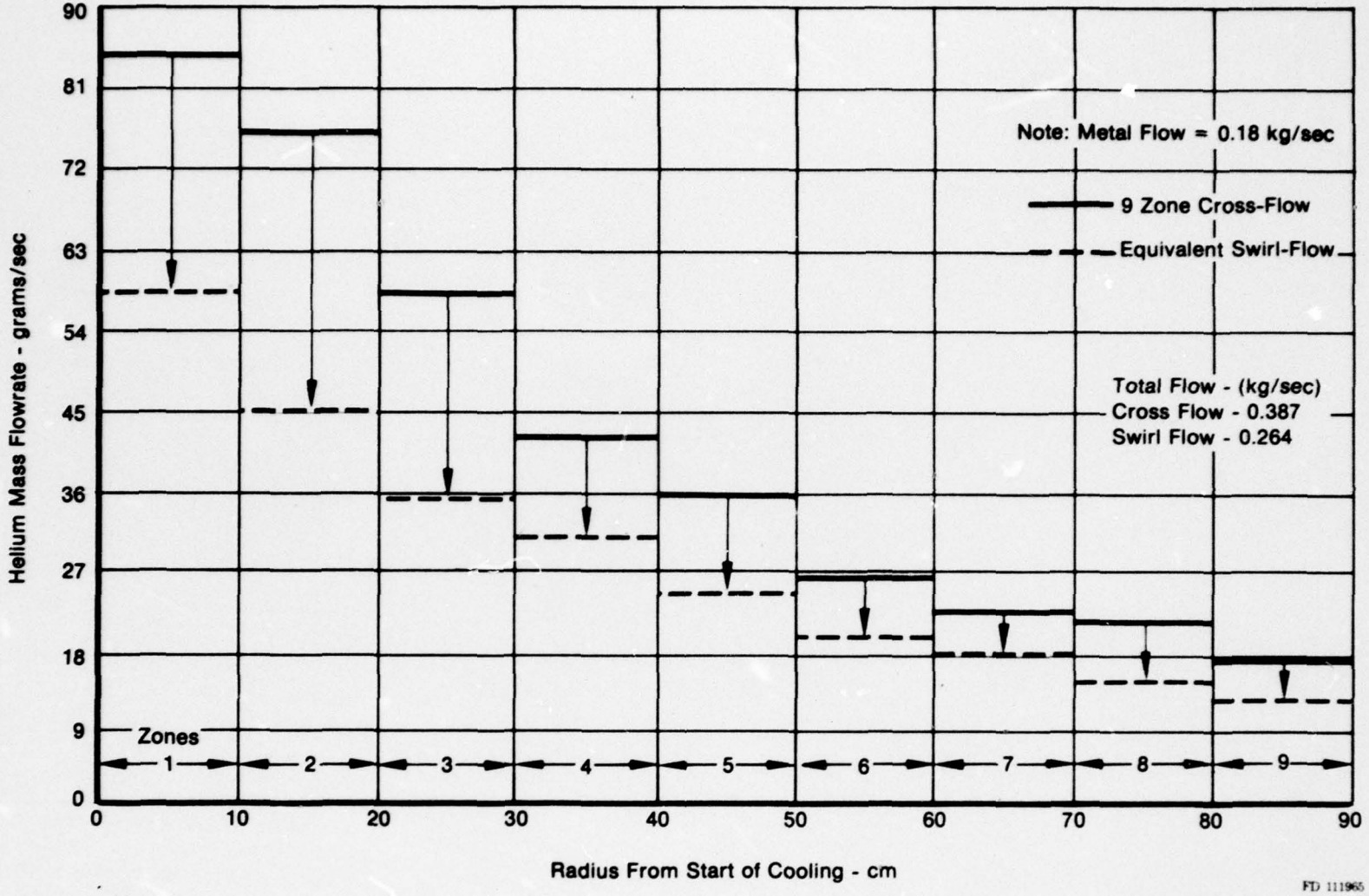

Figure 3. Comparison of Helium Throughput For Cross-Flow and Swirl-Flow Cooling

SECTION III
MATERIAL EVALUATION

Of the 20 runs made during this period, 15 were of IN-100 and MAR M-200 alloy and 5 were experimental compositions in which the carbon and refractory element concentrations were varied from the base IN-100 composition. Six of the lots of material were used in conjunction with previous powder runs to establish effective working parameters for compaction, extrusion, and forging. The balance of material was stored pending results of this investigation and is now in varying stages of containerization, consolidation, etc.

With respect to analysis of the powder, relatively little new information was obtained during this period. Each lot was evaluated in the manner reported previously and results continue to show the same features, suppression of secondary phases, high degree of chemical homogeneity, etc.

The first successful attempts to analyze the powders by scanning transmission electron microscopy were completed and results are presently being collated for the various structures of powder identified to date. The occurrence of a microcrystalline form continues to be observed. Positive identification that this is, in fact, a microcrystalline form was obtained through the use of electron channelling images. Figure 4 shows the difference in structure between the dendritic and microcrystal forms. It is very evident in this comparison that the dendritic form leads to a coarse grain (relative basis) particle whereas the microcrystal is, indeed, an aggregate structure of very fine grains.

As discussed in the previous two quarterly reports, one of two conditions is thought to be responsible for this change in the solidification mode. The first is that a rheocasting type phenomenon takes place on the atomizer itself, whereby fine dendrites form on the surface prior to atomization and are swept away subsequently into the quench media by residual liquid metal. The second is that critical undercooling takes place with subsequent homogeneous nucleation.

Until recently, a rheocast condition seemed to be the most likely cause of the solidification mode. This was based on observations of micros after prolonged etching in which ghost dendrite patterns seemingly appeared; on the fact that the atomizer acts as a highly efficient mixing device and could promote two phase atomization; and on measurements which suggested that the mean microcrystal diameter did not vary appreciably with particle size, and thus was somewhat independent of cooling rate.

The circumstances of undercooling were derived from results of microprobe analysis which showed the microcrystal form to be more homogeneous, and on the fact that initial cooling on the atomizer was taking place by conduction at very high heat transfer (wetted surface).

The lack of variation of mean microcrystal diameter with particle size in the initial studies were, indeed, negative to this latter belief. However, in the studies conducted with material produced in more recent months (when it can be safely stated that the device was operating in an overall stable mode), variations in mean diameter are very evident in particles of different sizes. Figure 5 shows the distinction. For the coarse particle sizes shown (about 50 μm), the mean diameter is about 2.2 μm. In the fine particles (about 25 μm), the mean diameter is about 0.7 μ m, only 1/3 that of the coarse. Under these circumstances, then, it becomes plausible to believe that critical undercooling could be the principal cause for the observed microstructural form.

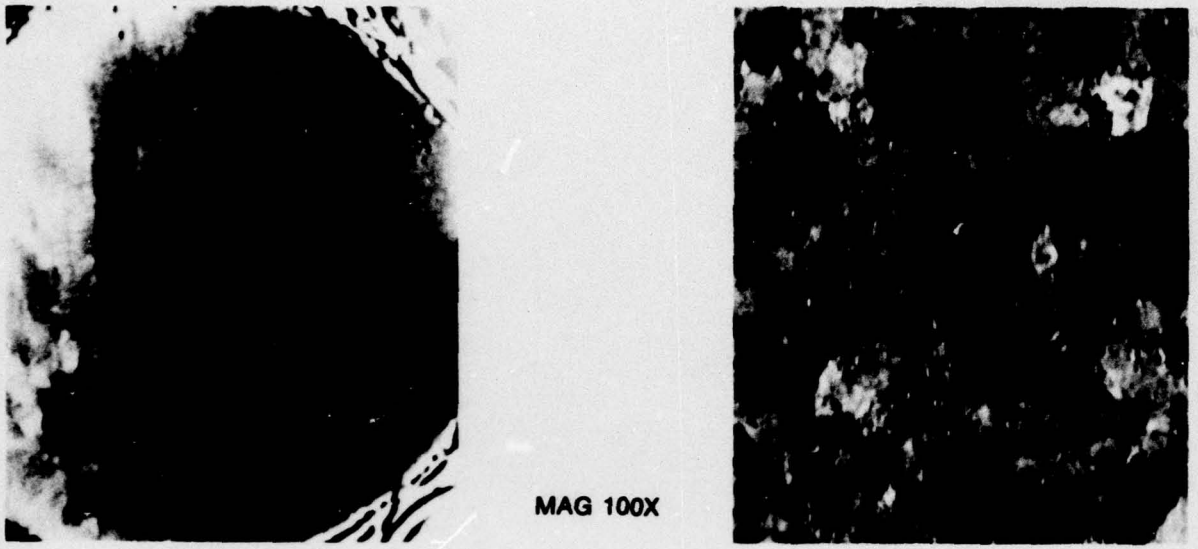

Figure 4. Structural Forms of Rapidly Solidified Powder

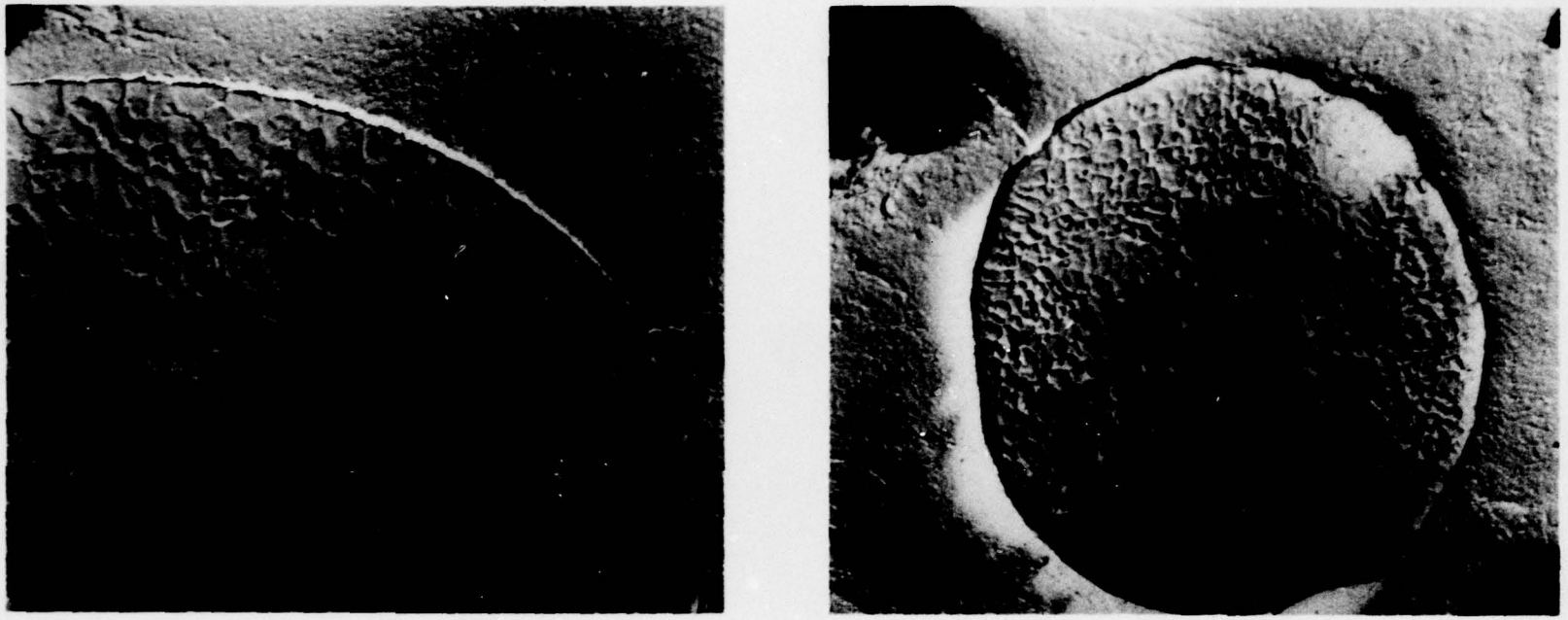

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Figure 5. Microcrystalline Variations in Mar M-200 Alloy Powder

Previous attempts to consolidate and extrude the fast quenched powders were based exclusively on parameters developed for conventional powder. These trials were unsuccessful, generally, in that only limited recrystallization after working was achieved and response to subsequent forging and heat-treatment was poor. Corrective actions were taken during this period and results to date have proven favorable. Two major changes were made for this purpose. The first was to abandon the use of consolidation within the extrusion press itself (using a blanked-off orifice) and the second was to modify the extrusion conditions by changing the die angle from 60 to 90 deg and by increasing the reduction from a nominal 6/1 to 8/1.

Hot isostatic pressing and isothermal compaction under a uniaxial load are now being used for compaction. Both produce favorable results. Although only several of many parameters for each process have been investigated, it appears more practical for our purpose to rely on hot isostatic pressing as the consolidation means. This method eliminates the need for appendage tooling, simplifies separation of the experimental material from its original container, and operates to equivalent time, temperature, and pressure parameters as does the isothermal technique.

Typical of the compacted form now being obtained is shown in figure 6. This was an isothermal compaction of MAR M-200 alloy at 1065°C. Recrystallization was achieved locally, and bonding is complete. It is interesting to note that only particles displaying dendritic solidification patterns remain essentially underformed. No spurious reactions were observed on particle surfaces for any of the lots of material evaluated.

Extrusions of the compacted material were run for us by the Air Force Materials Laboratory and pertinent data for each are listed in Table I. All runs produced sound bar stock with no visual defects. Oxygen samples showed also that acceptable O_2 levels were achieved for each, typically in the range of 50 ppm. Figures 7 and 8 show the microstructures of IN-100 and MAR M-200 alloy after extrusion and after forging at the temperatures indicated. Unlike the previous attempts, the material exhibited internal soundness, and recrystallization was complete after forging. The forgings were run under isothermal GATORIZING™ parameters and, like the extrusions, had no surface defects.

The IN-100 material is being tested under conditions whereby a direct comparison to existing data could be made. After forging, the material was heat-treated to wrought IN-100 alloy specifications and tested in tensile, creep, creep-rupture, and low-cycle fatigue. The results, to date, are shown in Table II. The goal listed for each condition is the average property of the presently used powder IN-100 material. Tensile properties appear similar to existing material. Stress-rupture, creep, and low cycle fatigue all look better. The data are limited, obviously, and must be tempered because of this. Additional tests are in progress, however, and more are planned as material becomes available, so that a reasonably good comparison should be available in the next report period.

The MAR M-200 alloy was tested in a directionally recrystallized condition. The appearance of the annealed forgings is shown in figure 9. Subsequently, the material was aged to the same specification criteria as the IN-100 alloy and tested in creep-rupture at conditions depicting turbine airfoil requirements. The results are listed in Table III and the goals given here are the average times for directionally solidified MAR M-200 (+Hf) alloy, our designation is PWA 1422. The data appear about equal. Creep resistance at 1800°F looks promising but, again, the sampling size is small. As with IN-100, more testing is planned.

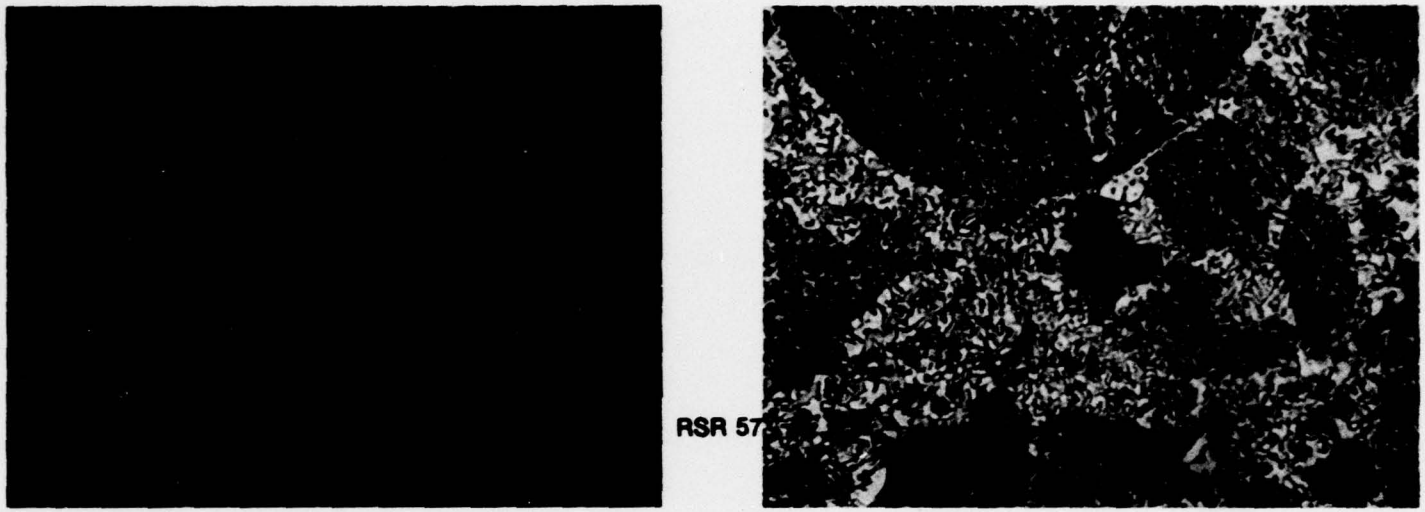

Figure 6. Microstructure of Compacted Mar M-200 Alloy

Table 1. Consolidation and Extrusion Operations

Alloy ID	Type	Temperature °C	Temperature °F	Pressure ksi	Pressure Pa × 10⁹	Time hr	Extrusion Temperature °C	Extrusion Temperature °F	Reduction
IN100-39	HIP	1218	2225	15	1.03	8	1065	1950	~8:1
IN100-40	HIP	1218	2225	15	1.03	8	1065	1950	~8:1
CoTaC-52	Isothermal	1065	1950	20	1.37	4	1204	2200	~8:1
MAR M-200-56	Isothermal	1065	1950	20	1.37	4	1177	2150	~8:1
MAR M-200-57	Isothermal	1065	1950	25	1.72	6	1121	2050	~8:1
MAR M-200-58	Isothermal	1065	1950	25	1.72	4	1121	2050	~8:1
MAR M-200-58A	Isothermal	1065	1950	25	1.72	4	1149	2100	~8:1
MAR M-200-59	Isothermal	1065	1950	25	1.72	4	1149	2100	~8:1
MAR M-200-60	Isothermal	1065	1950	25	1.72	4	1121	2050	~8:1
MAR M-200-60A	Isothermal	1065	1950	25	1.72	4	1149	2100	~8:1
MAR M-200-61	Isothermal	1065	1950	25	1.72	4	1121	2050	~8:1

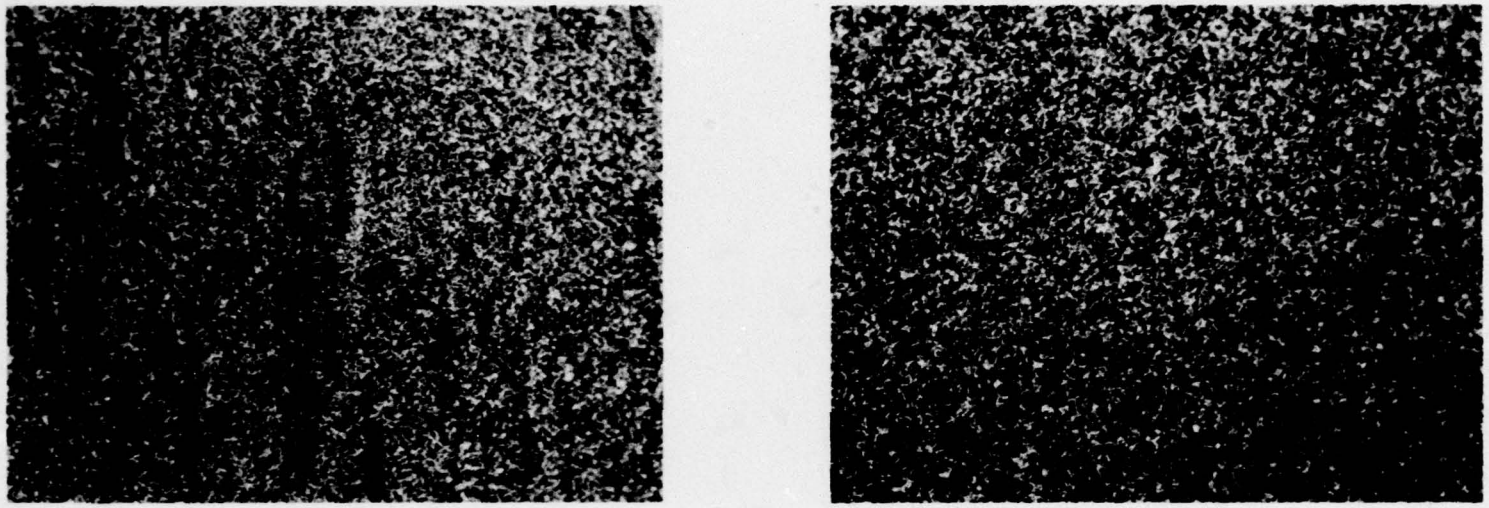

Figure 7. Microstructure of Extruded and Extruded and Forged IN-100

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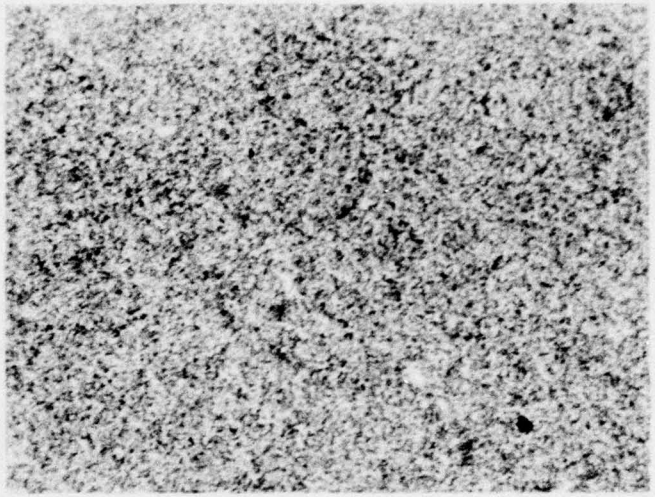
Extruded 8/1 at 1121°C

RSR 57



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After Forging at 1171°C

RSR 58

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Figure 8. Microstructure of Extruded and Extruded and Forged Mar M-200

Table 2. Mechanical Properties of RSR IN-100 Alloy

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ID	Temperature C	Temperature F	0.2% YS ksi	0.2% YS MPa	UTS ksi	UTS MPa	El %	RA %
40-3C	Ambient		162.3	1119.0	229.0	1578.9	23.0	24.0
39-4			162.5	1120.4	229.0	1578.9	28.0	28.0
40-30			162.3	1119.0	230.1	1586.5	24.0	24.0
Goal			163.0	1123.8	230.0	1585.8	22.0	22.5
40-40	704	1300	159.6	1100.4	182.8	1260.4	13.0	12.5
39-40	704	1300	159.1	1097.0	182.3	1256.9	16.0	15.7
40-3	704	1300	160.0	1103.2	179.6	1238.3	14.0	14.7
Goal	704	1300	158.0	1089.4	184.0	1268.6	18.5	17.0

Stress - Rupture
732°C, 637.8 MPa
(1350°F, 92.5 ksi)

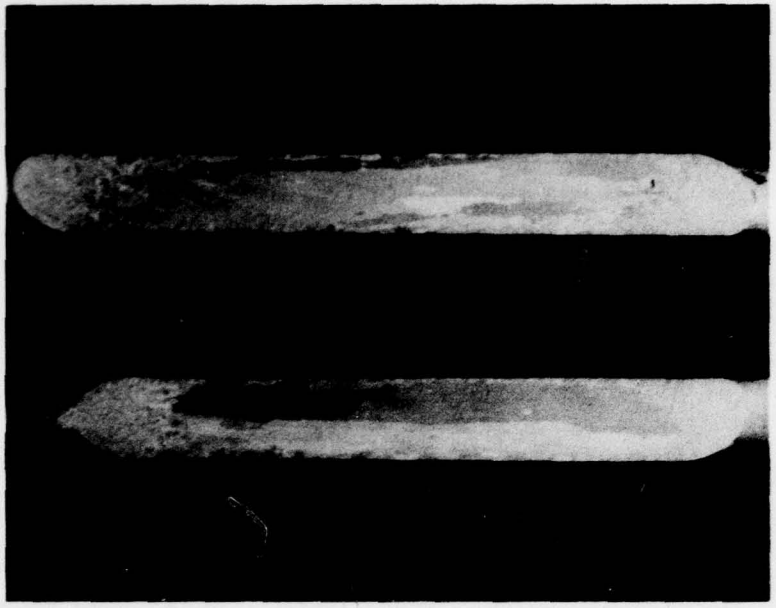
ID	Failure, hr	El, %	RA, %
39-3	83.7	8.3	16.1
40-4C	91.6	9.7	16.9
39-3 Notch	104.8	-	-
40-40 Notch	86.5	-	-
Goal	34.0	-	-

Creep
704C, 551.6 MPa
(1300°F, 80 ksi)

ID	0.1%, hr	0.2%, hr
40-2A	169.4	289.4
Goal	138.0	195.0

Low-Cycle Fatigue
538C (1000F), 1% Total Strain

ID	Cycles to Failure
39-2.1	15,830
Goal	6,000



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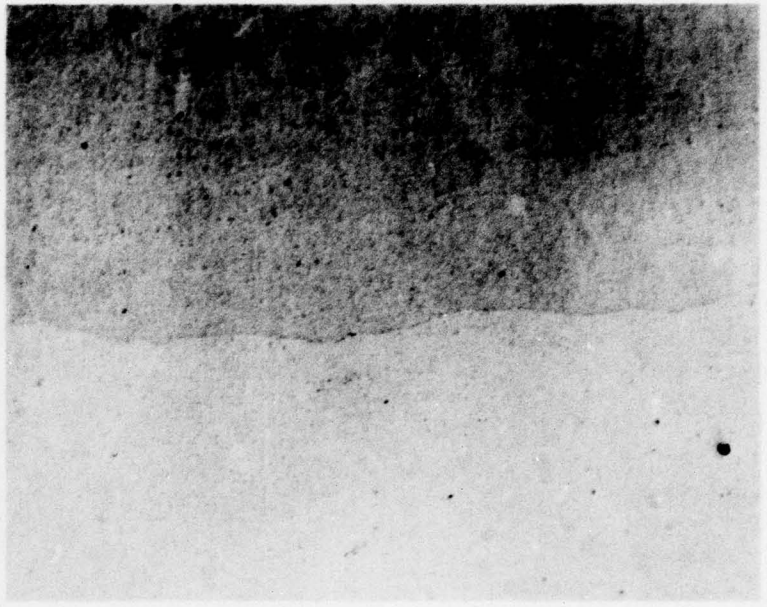
Cross-Section of Forging



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Grain Boundary Region

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Figure 9. Grain Growth in Mar M-200 Alloy

Table 3. Creep-Rupture Properties of RSR MAR M-200 Alloy

	Temperature		Stress		1%	Failure	El
ID	°C	°F	ksi	MPa	Hour	Hour	%
57-5.1	760	1400	95	655	163.0	179.6	9.1
57-5.2*	760	1400	95	655	37.5	45.4	19.0
Goal	760	1400	95	655	120.0	170.0	-
57-5.3	982	1800	31.5	217.2	41.8	54.5	15.1
57-5.4	982	1800	31.5	217.2	24.5	67.9	12.9
Goal	982	1800	31.5	217.2	19.0	55.0	-

*Substandard sample

The alloy study has taken on fairly large proportions of the total program effort at this time. The consolidation parameter study has been expanded for the superalloy powders processed to date, heat-treat studies have been initiated to ensure that our selection of precipitation-aging cycles are adequate, and potential reactivity of the new alloys is being evaluated.

As part of this latter evaluation, we have included a screening procedure for experimental compositions based on laser surface melting to achieve locally rapid solidification. This method permits analysis subsequently to determine whether sought after elemental features were achieved without the need to run a large quantity of powder. Cooling rates by laser surface treatment can be made to duplicate those in the powder device. A typical laser treated sample is shown in figure 10. The surface treated area is generally representative of 10^6°C/sec cooling. The particular sample shown had a high Ti and Al concentration, relative to existing alloys, and a carbon concentration about triple that which is now common. The sample shows that rapid solidification can produce the homogeneity needed to effectively utilize these elements at the concentrations of interest. Twenty-five of these samples have been run and are now undergoing evaluation for the purpose of selecting alloys for atomization. The CoTaC alloy and the other experimental alloys already converted into powder are being processed at various stages for future testing. Results from these, as well as the additional testing of the base materials, should be available in the next report period.

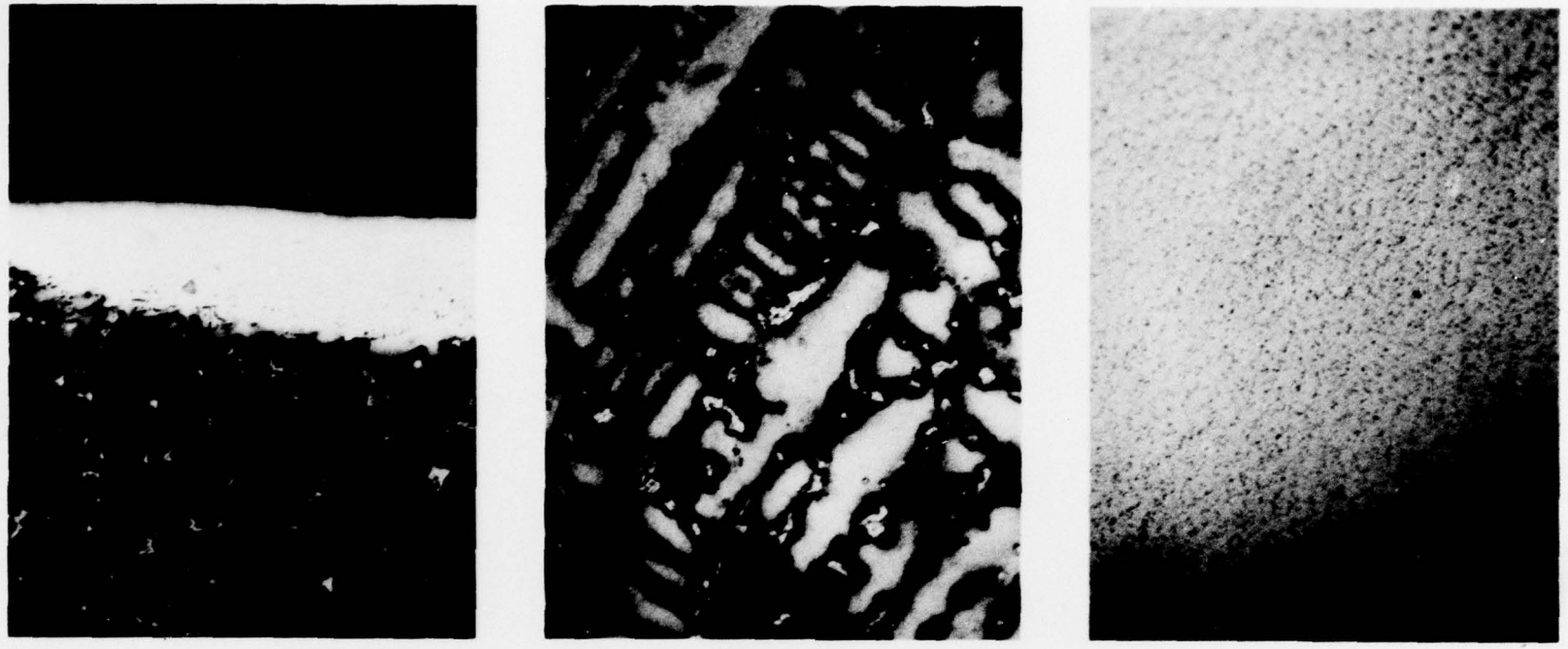

Figure 10. Rapid Solidification By Laser Surface Melting